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For presentation to the Chemistry Department of the Air Force Academy, Colorado Springs, CO, 12 April 2012.

14. ABSTRACT

In this presentation, structures were demonstrated to be reactive towards a variety of dichlorosilanes. Solubility of F-POSS compounds were shown to be influenced by chemical functionality. Functionality was shown to be influential on contact angle measurements. Scientists are currently working on other monomers and polymers for F-POSS. F-POSS compounds have a near limitless potential in producing a variety of new hydrophobic, oleophobic, or omniphobic polymer composites.

15. SUBJECT TERMS

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THE AIR FORCE RESEARCH LABORATORY
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Synthesis and Free Radical Polymerization of Fluorinated Polyhedral Oligomeric Silsesquioxane (F-POSS) Macromers: Precursors for Low Surface Energy Materials and Devices

Sean M. Ramirez

ERC Inc., Air Force Research Laboratory

Propulsion Directorate

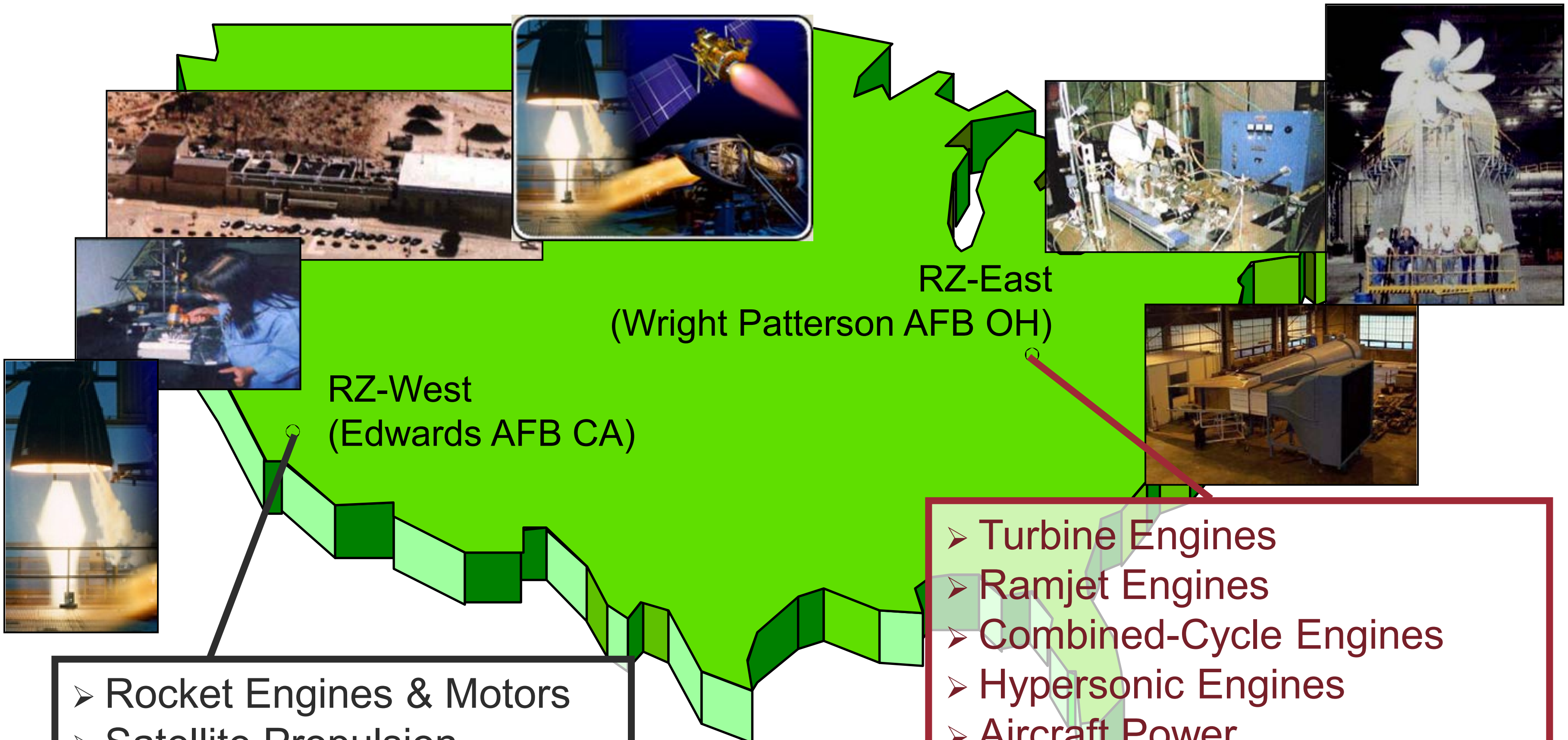
Edwards Air Force Base, CA

April 2012

AFRL Propulsion Directorate

(AFRL/RZ)

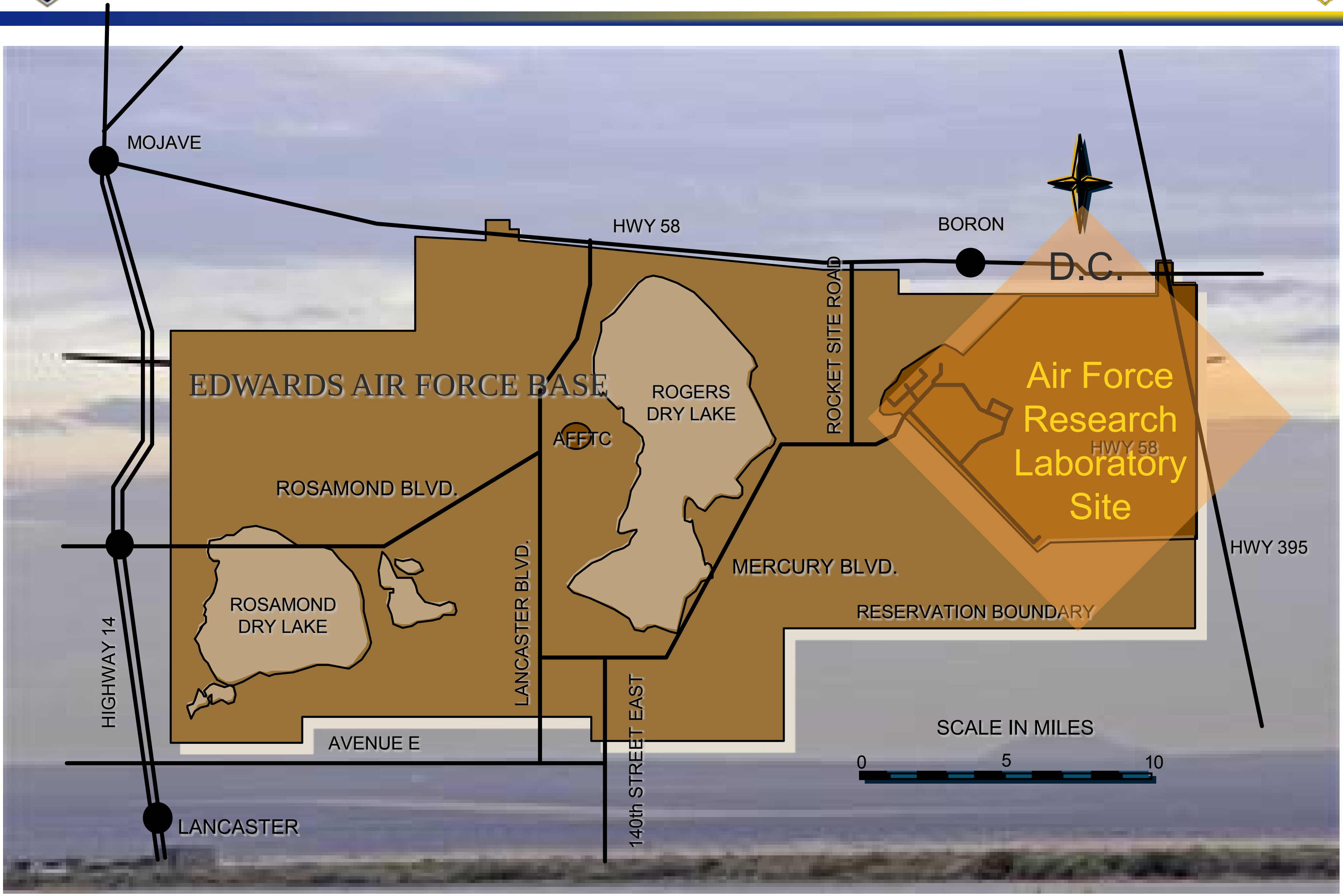
AFRL Propulsion Directorate



- Rocket Engines & Motors
- Satellite Propulsion
- Advanced Propulsion
- Fuels and Propellants
- System Analysis

- Turbine Engines
- Ramjet Engines
- Combined-Cycle Engines
- Hypersonic Engines
- Aircraft Power
- Thermal Management
- Fuels and Propellants
- System Analysis

Edwards AFB


MOJAVE
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Laboratory
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ROSAMOND
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RESERVATION BOUNDARY
HIGHWAY 14
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140th STREET EAST
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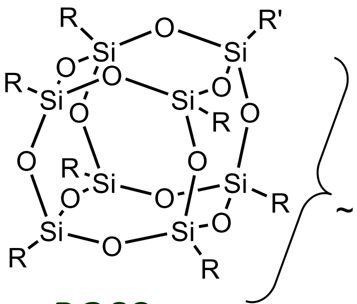
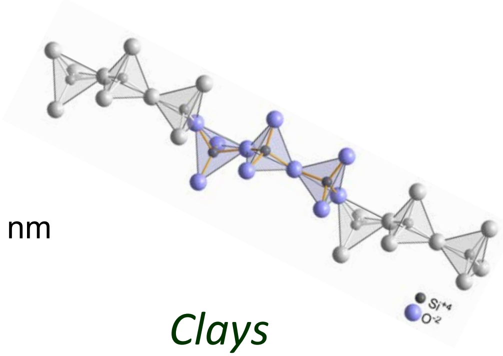
Basic and Applied Research

Basic Research



~1 nm

POSS



Clays

- Basic POSS research funded by AFOSR

Solid Rocket Motors

- Polymers are >50% of inert mass
- Nanocomposites allow significant weight reduction
- Transition target - Boost, strategic, & tactical SRMs
- Use possible in other Air Force applications

Atlas V

Liquid Rocket Engines

- Fluorinated polymer nanocomposites
- Reduce cost and improve performance
- Most hydrophobic crystalline solids known
- Superoleophobic surfaces produced
- Fuel separation

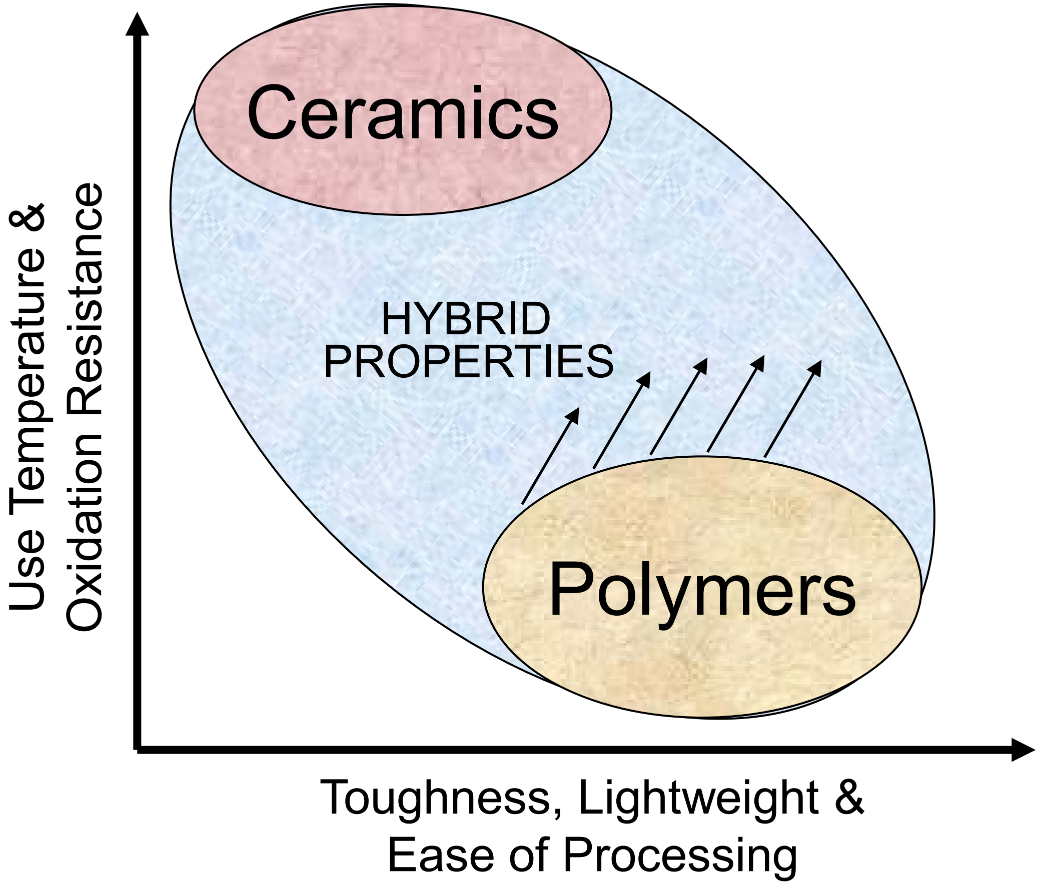
Liquid Demo Engine

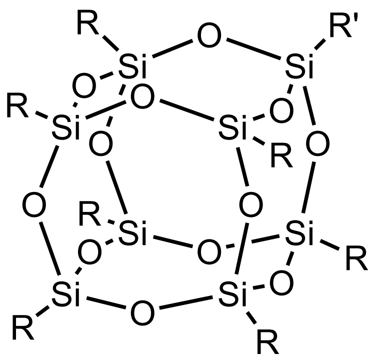
Polymer Matrix Composites

- Replace metals for mass reduction
- Resistance to oxidation seen in nanocomposite PMCs
- Developing PMC resins for high-temperature applications
- Moisture uptake properties improved with added nano-materials

Bushings

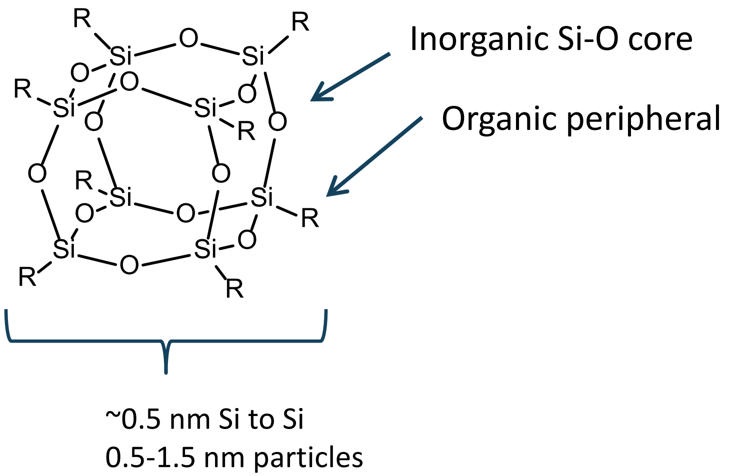
Hybrid Inorganic/Organic Polymers





POSS $(RSiO_{1.5})_n$

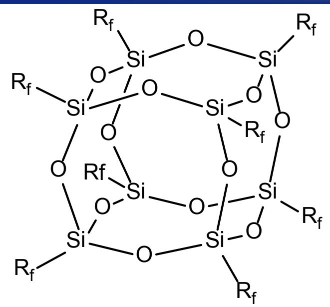
- Organic-inorganic framework
- Well-defined, 3-D nanostructure
- Can carry *functional* groups
- Thermally and chemically robust
- Used in thermoset and thermoplastic polymers, temperature nanocomposites, coatings, surface modifiers, and many other applications



Cordes, D. B.; Lickiss, P. D.; Rataboul, F. *Chem. Rev.* ***2010****, 110, 2081.*

Phillips, S. H.; Haddad, T. S.; Tomczak, S. J. *Current Opinion in Solid State and Materials Science* ***2004****, 8, 21.*

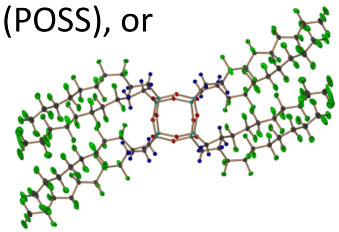
Introduction to F-POSS



$R_f = -(CH_2)_2(CF_2)_7CF_3$

(1,1,2,2-tetrahydroperfluorodecyl)$_8Si_8O_{12}$ Polyhedral Oligomeric Silsesquioxane (POSS), or fluorodecyl POSS

- hybrid organic-inorganic structure
- well-defined polyhedral architecture
- long-chain fluoroalkyl substituents on periphery of cage

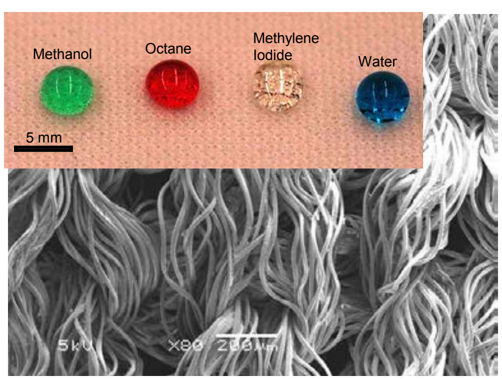


Due to its unique structure, fluorodecyl POSS has one of the lowest surface energies of any crystalline solid currently known

- fluorodecyl POSS	9.3 mN/m
- polytetrafluoroethylene	18-20 mN/m
- CF_3 monolayer	6.7 mN/m

Low surface energy and other unique properties of fluorodecyl POSS has enabled the development of various types of tunable non-wetting polymeric surfaces

Superhydrophobic/oleophilic dip-coated fabric
Tuteja *et al*, Science, **2007**, 318, 1618

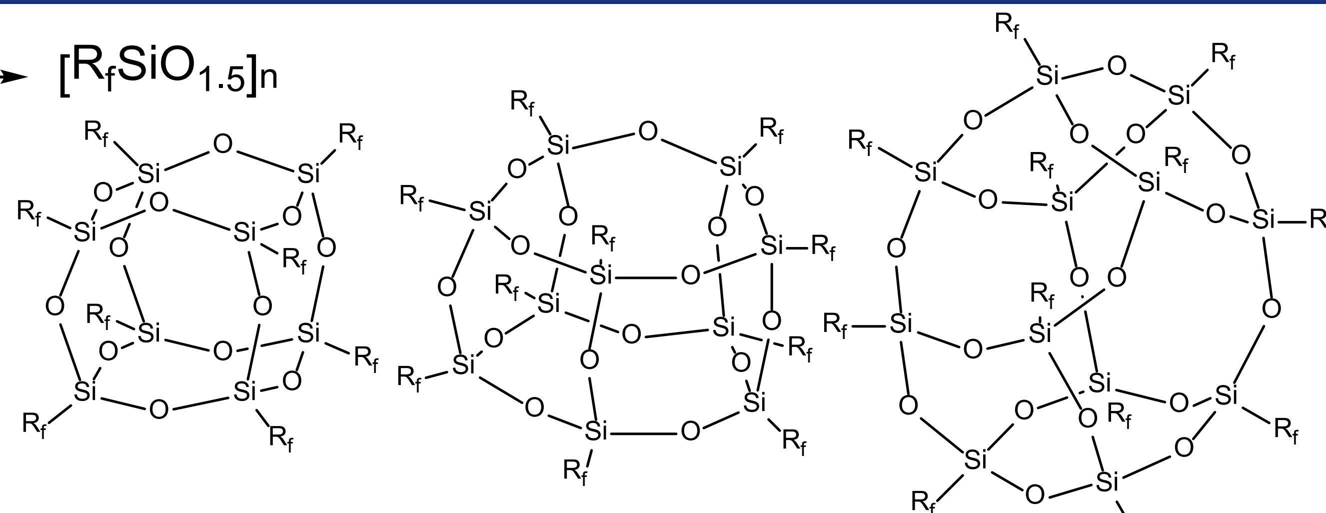


Superamphiphobic dip-coated fabric
Choi *et al*, Adv Mater, **2009**, 21, 2190

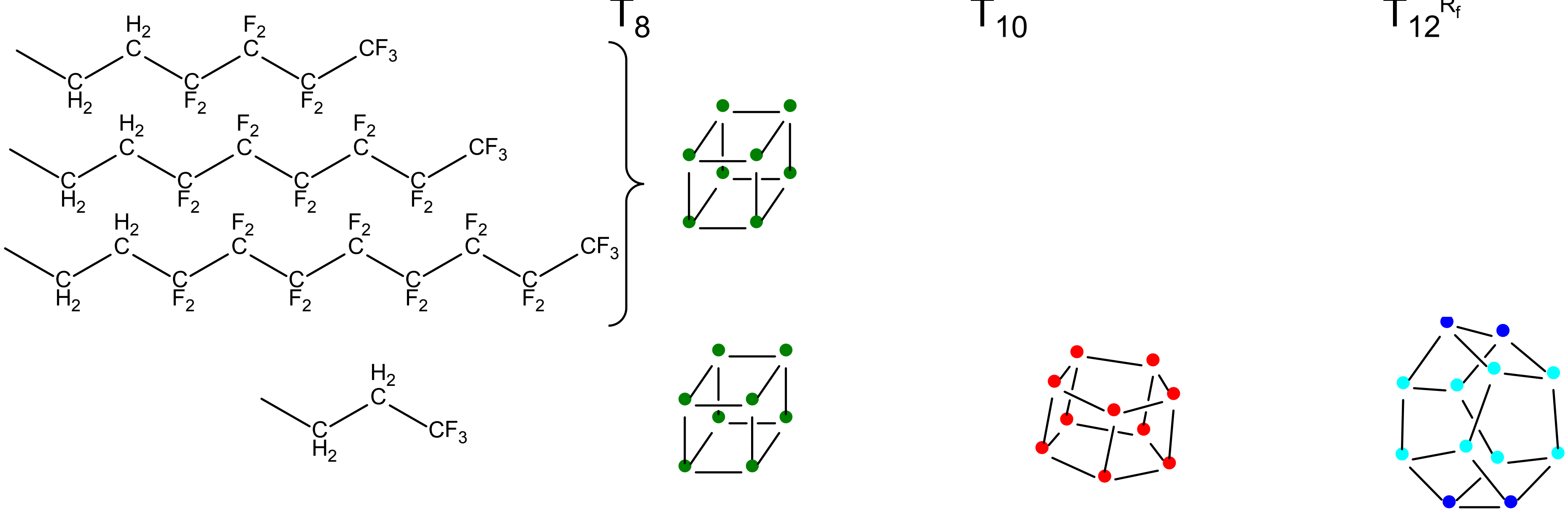
Superamphiphobic electrospun surfaces
Tuteja *et al*, PNAS, **2008**, 105, 18200

Synthesis of F-POSS

$$R_fSi(OEt)_3 \xrightarrow[EtOH]{KOH/H_2O} [R_fSiO_{1.5}]_n$$

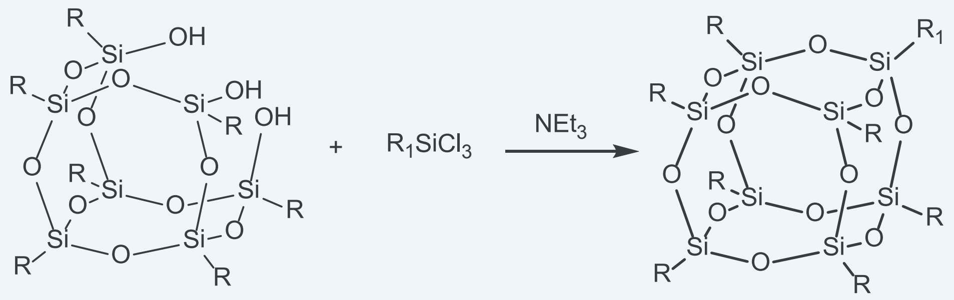


T_8 T_{10} T_{12}



Functional F-POSS (Open-Caged)

- Close-caged structures are accessible and have proven versatile in polymer composites
  - Limitations
    - Solubility, mechanical robustness (surface abrasion), no sites for functionality
- Open-caged structures would allow for functionalization of F-POSS
  - Open door for use a *building block* material for *low surface energy materials*
- Applications
  - Mechanical robust superhydrophobic/oleophobic/omniphobic surfaces
    - Via covalently attached F-POSS to substrate (surface, nanoparticle, polymer matrix)
  - Effects on polymer composite properties
    - Wetting, phase behavior, solubility, etc….



- Open cages lead to functional POSS structures
- Reactions are simple
- High yields typically reported

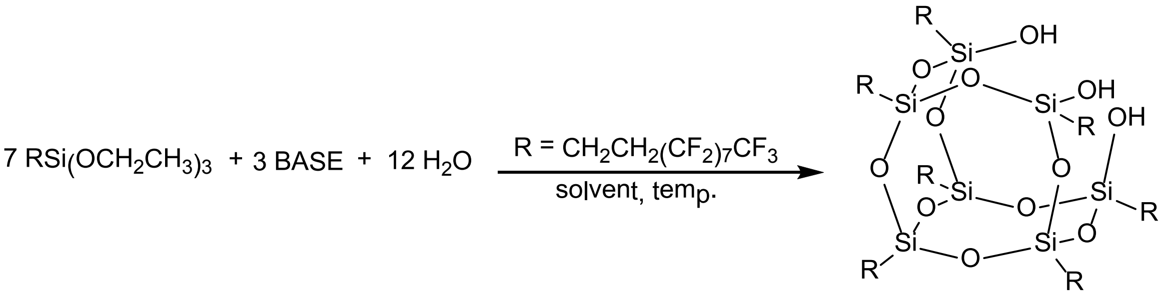
Methods to Produce Incompletely Condensed Silsesquioxanes

- Bottom-up approach
  - Acid or base mediated from $RSiCl_3$ or $RSi(OR)_3$
  - Condensation reaction
  - Balance of stoichiometry, temperature, reaction time, patience, and luck
  - Stopping POSS synthesis early, before cages closes
  - More common approach
- Top-down Approach
  - Strong acid or base mediated
  - Starting from a POSS cage
  - Conversion of Si-O-Si bonds to Si-O $^{(-)}$ C $^{(+)}$ or Si-OH bonds
  - Opening up POSS cage

Which method can be applied to F-POSS?

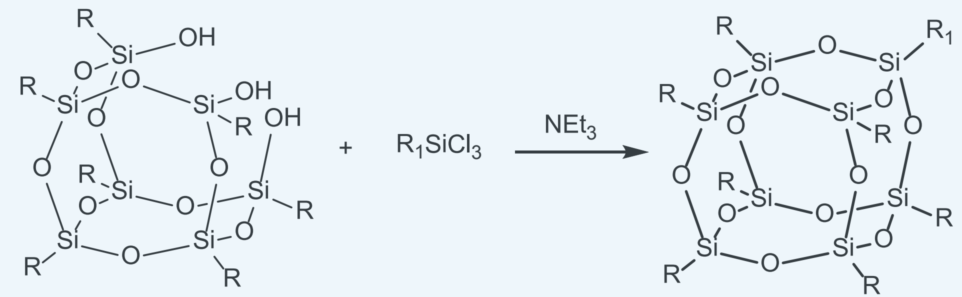
Feher, F. J.; Terroba, R.; Ziller, J. W. *Chemical Communications* ***1999***, 2309. Feher, F. J.; Newman D.A;. Walzer, J.M., *J. Am. Chem. Soc.*, **1989**, 111, 1741. Feher, F. J.; Soulivong, D.; Nguyen, F.; Ziller, J. W. *Angew.Chem. Inter. Ed.* ***1998**, 37,* 2663. Feher, F. J.; Soulivong, D.; Nguyen, F. *Chem. Commun.* ***1998**,* 1279.

Synthesis of F-POSS-$(OH)_3$



R = $CH_2CH_2(CF_2)_nCF_3$

- Synthesis discussed in patents*
- Works for trifluoropropyl groups
- Solubility is critical in this reaction
- Fluorinated compounds not miscible in most organics once condensation begins to occur
- ***Does not work for long-chain F-POSS***
- Tried under various conditions
  - Solvent, temperature, reaction time, base



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*Yamashita, Y.; Hayashi, K.; Ishihara, M.; (Mitsubishi Materials Corp., Japan; Dai Nippon Toryo Co., Ltd.). Application: JP, 2000; pp 12 pp. Yamashita, Y.; Hayashi, K.; Ishihara, M.; (Mitsubishi Materials Corp., Japan; Dai Nippon Toryo Co., Ltd.). Application: JPJP, 2000; pp 9 pp.

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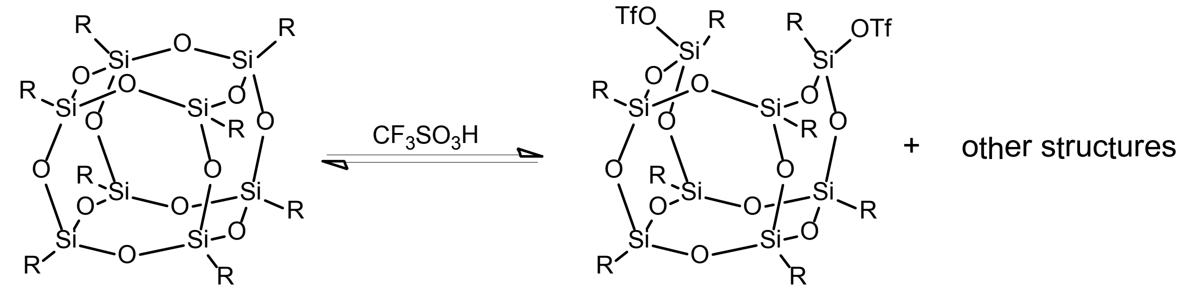
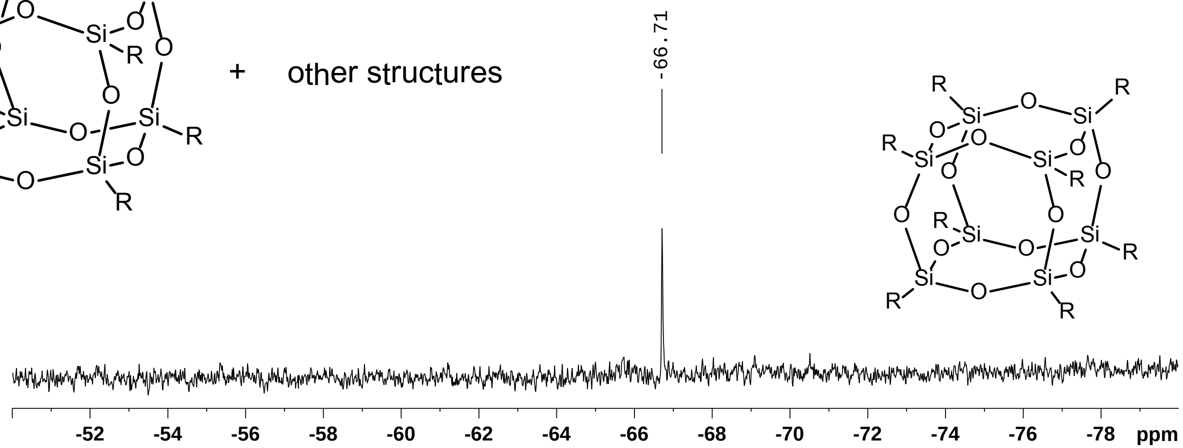
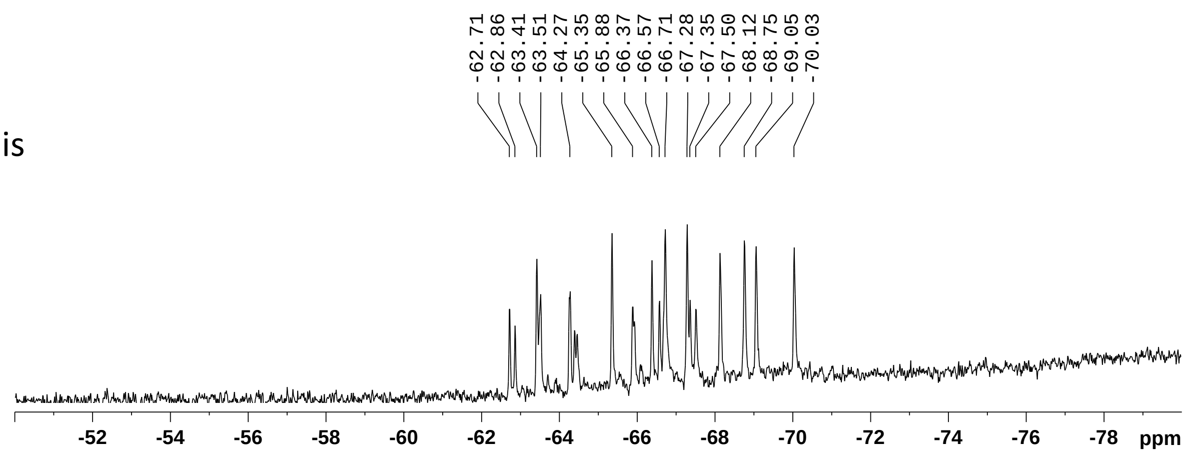
Initial Reactions with Triflic Acid



R = $CH_2CH_2(CF_2)_7CF_3$

- Fluorodecyl T_8
- Reacted with triflic acid to open cage structure
- Structures analyzed with ^{29}Si NMR
- Equivalents of triflic acid to POSS cage is important to success of reaction
- Disappointing results

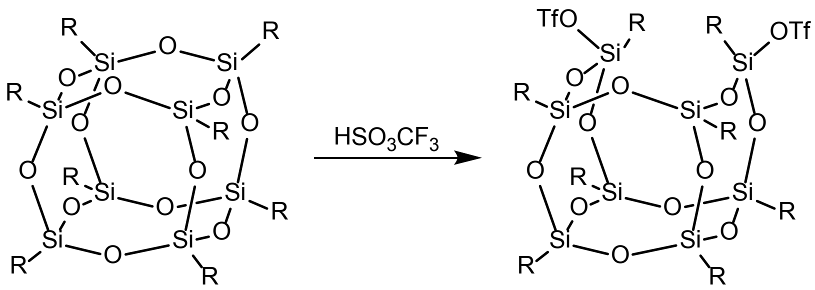




^{29}Si NMR taken in fluorinated solvent

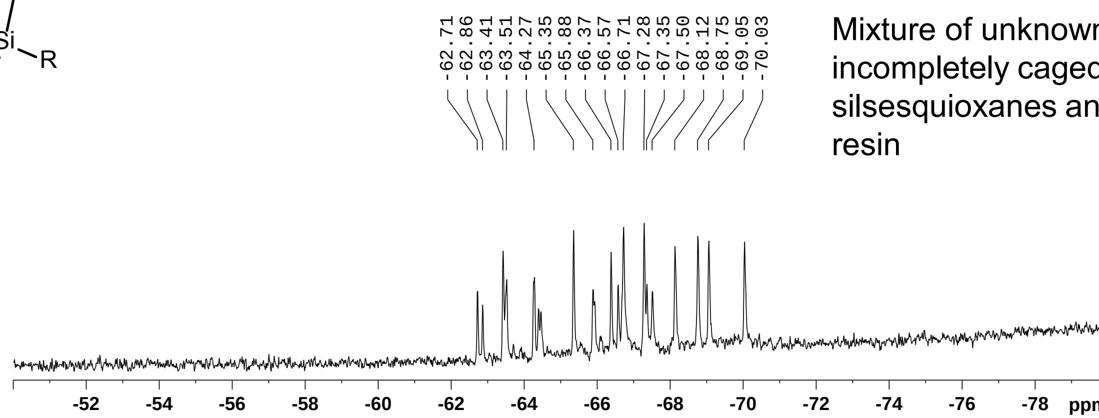
. Feher, F. J.; Soulivong, D.; Nguyen, F. *Chem. Commun.* ***1998,*** 1279.

Synthesis of F-POSS-(OTf)$_2$

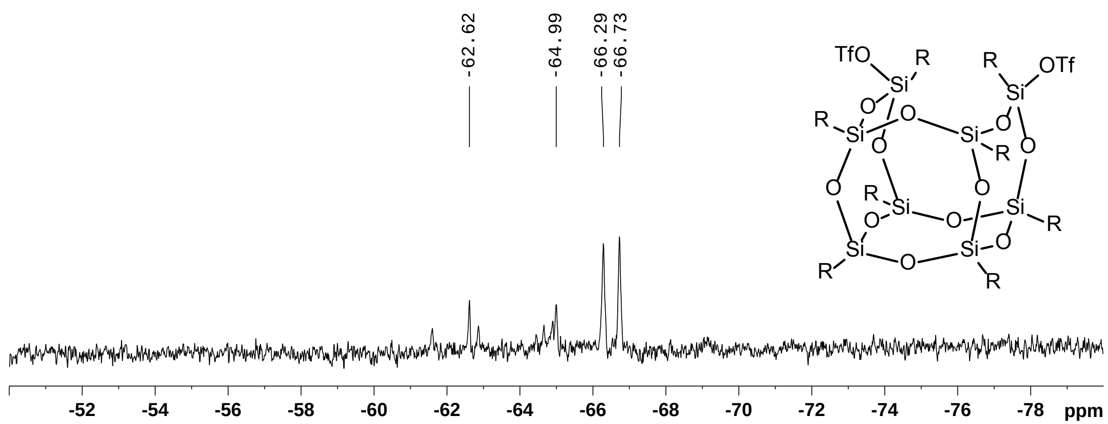


R = $CH_2CH_2(CF_2)_7CF_3$

- After a little bit of refining
- An open cage structure is partially visible
- Starting material is still present
- Reaction not very clean
- Si ratio (1:1:2)

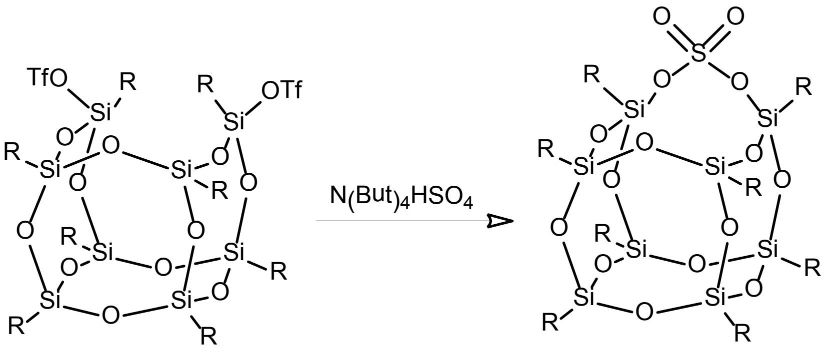


Mixture of unknown incompletely caged silsesquioxanes and resin



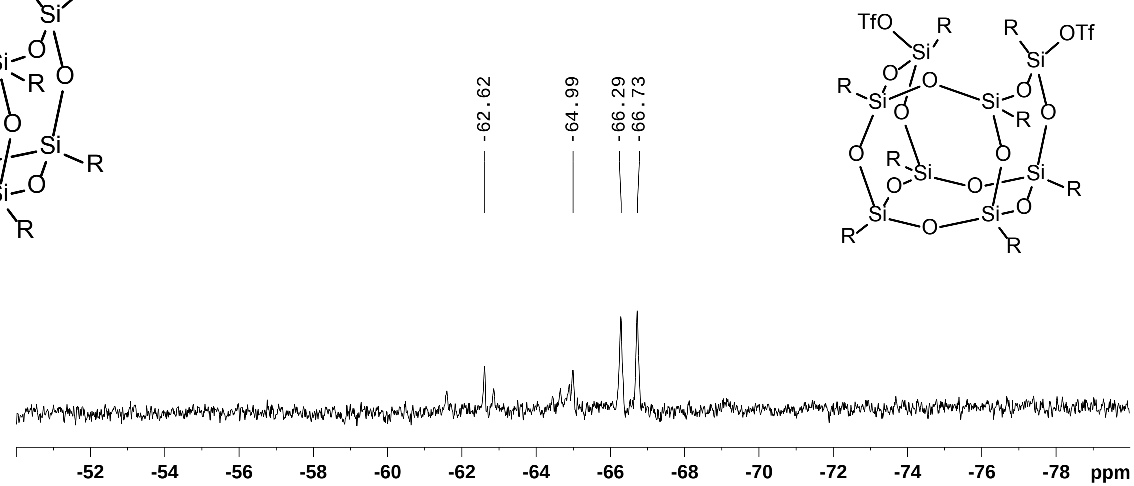
^{29}Si NMR taken in fluorinated solvent

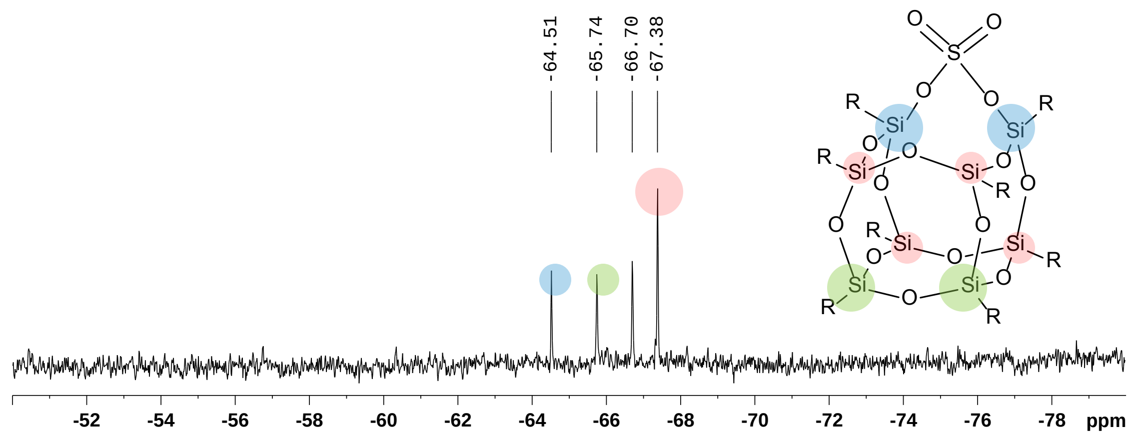
Synthesis of F-POSS-SO_2



R = $CH_2CH_2(CF_2)_7CF_3$

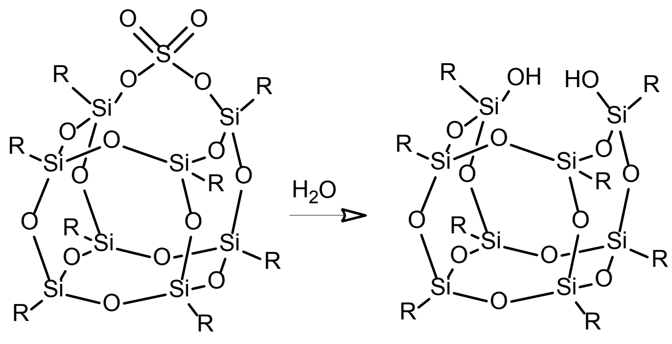
- Bridge sulfate cleans up reaction
- Structure significantly more stable than then F-POSS-$(OTf)_2$, however still difficult to isolate
- Removal of starting material extremely difficult
- Si ratio (1:1:2)



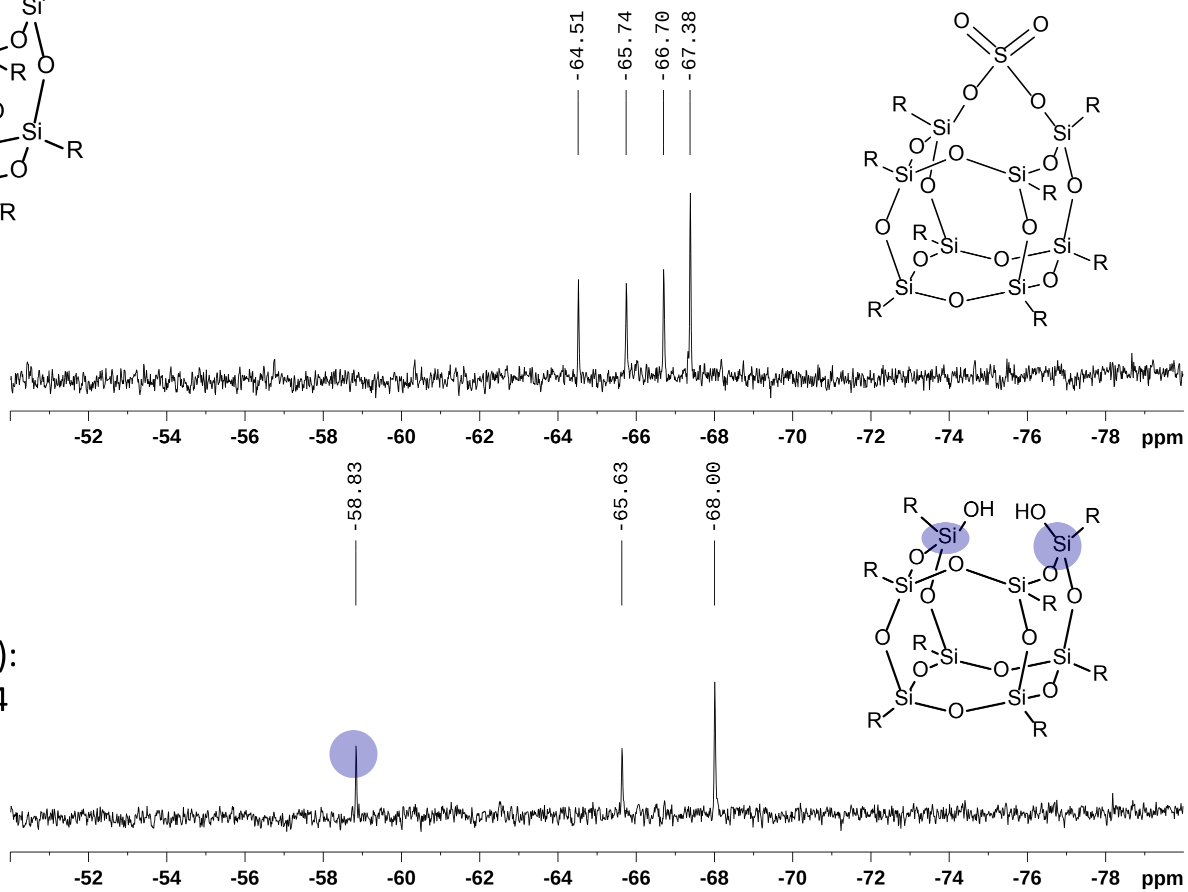


^{29}Si NMR taken in fluorinated solvent

Synthesis of F-POSS-$(OH)_2$



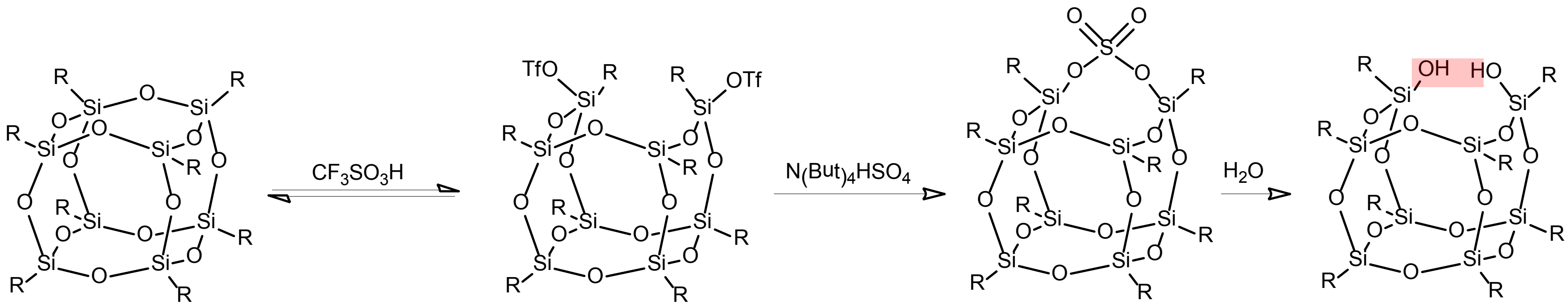
R = $CH_2CH_2(CF_2)_7CF_3$



^{29}Si NMR taken in fluorinated solvent

- Acidic conditions are used to remove the bridge sulfate complex
- Silanol peak at -58.8 ppm
- F-POSS-$(OH)_2$ is more stable than both F-POSS-$(OTf)_2$ and F-POSS-SO_2
- Anal. Calcd. for $C_{80}H_{34}F_{136}O_{13}Si_8$ (found): C, 23.94 (23.99), H, 0.85 (0.75), F, 64.44 (64.72)
- Dehydration of disilanol leads to T_8 formation
- Si ratio (1:1:2)

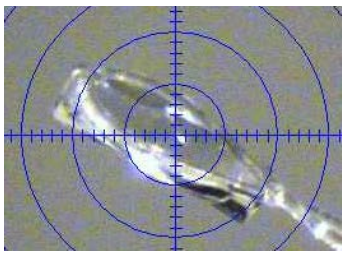
Incompletely Condensed Silsesquioxane

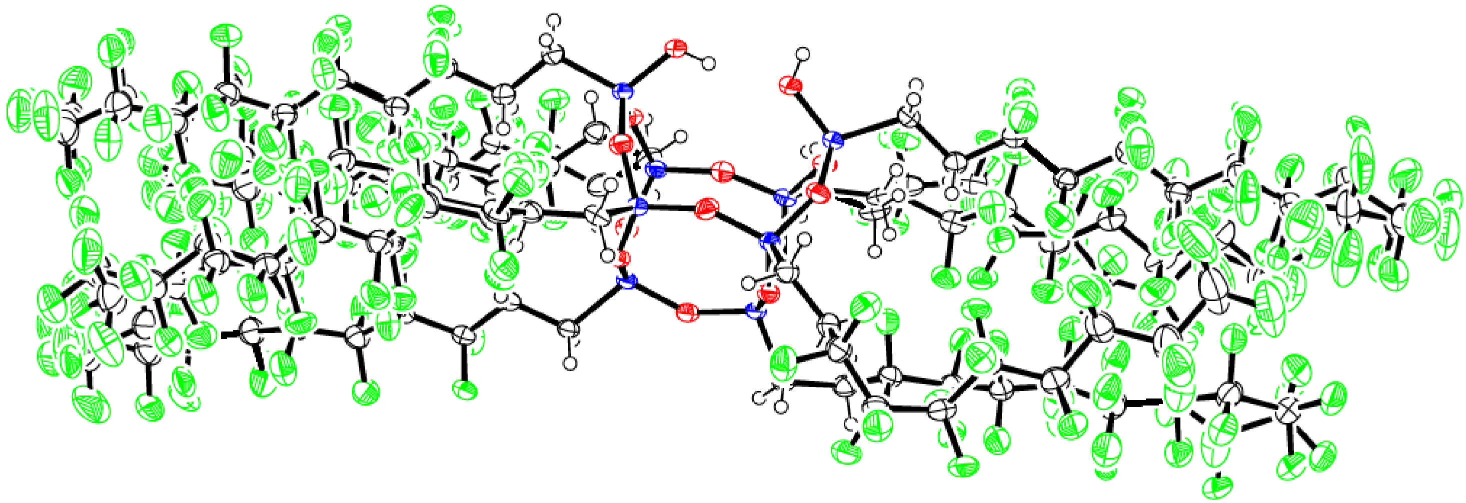


$R_f = CH_2CH_2(CF_2)_7CF_3$

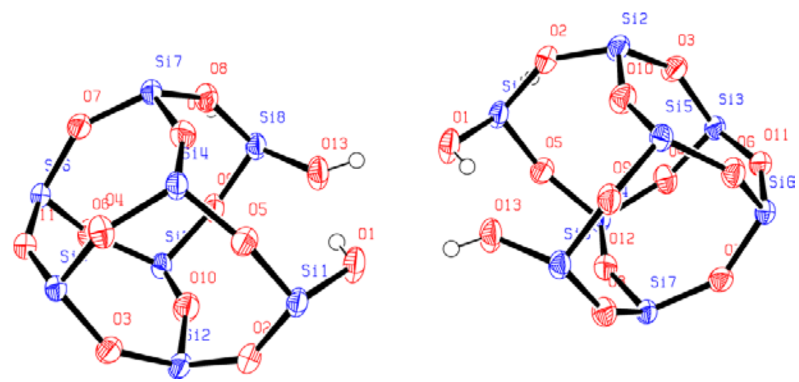
Ramirez, S. M.; Diaz, Y. J.; Campos, R. ; Stone, R.T.; Haddad, T.S.; Mabry, J.M., *J. Am. Chem. Soc.,* **2011**, *133*, 20084.

X-Ray Crystal Structure of Disilanol



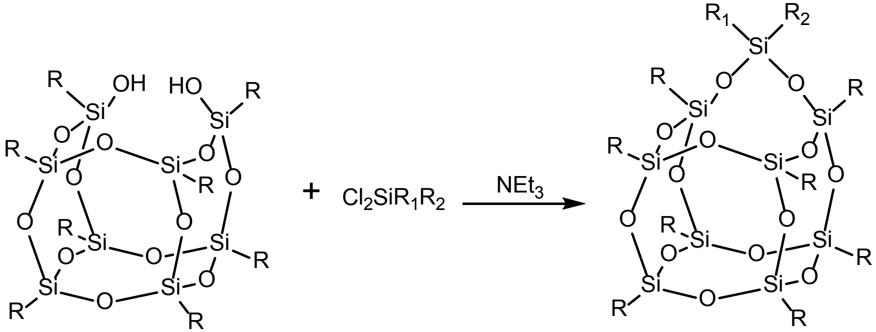


- Crystal structure is dimeric via intra- and intermolecular hydrogen bonding between silanols.
- M_r=,monoclinic, space group P2(1)/c , a=11.84(10) Å, b=57.11(6) Å, c=19.06(2) Å, α= 90.00°, β=92.21(10) °, γ=90.00 °, V= 12878(2) Å^3



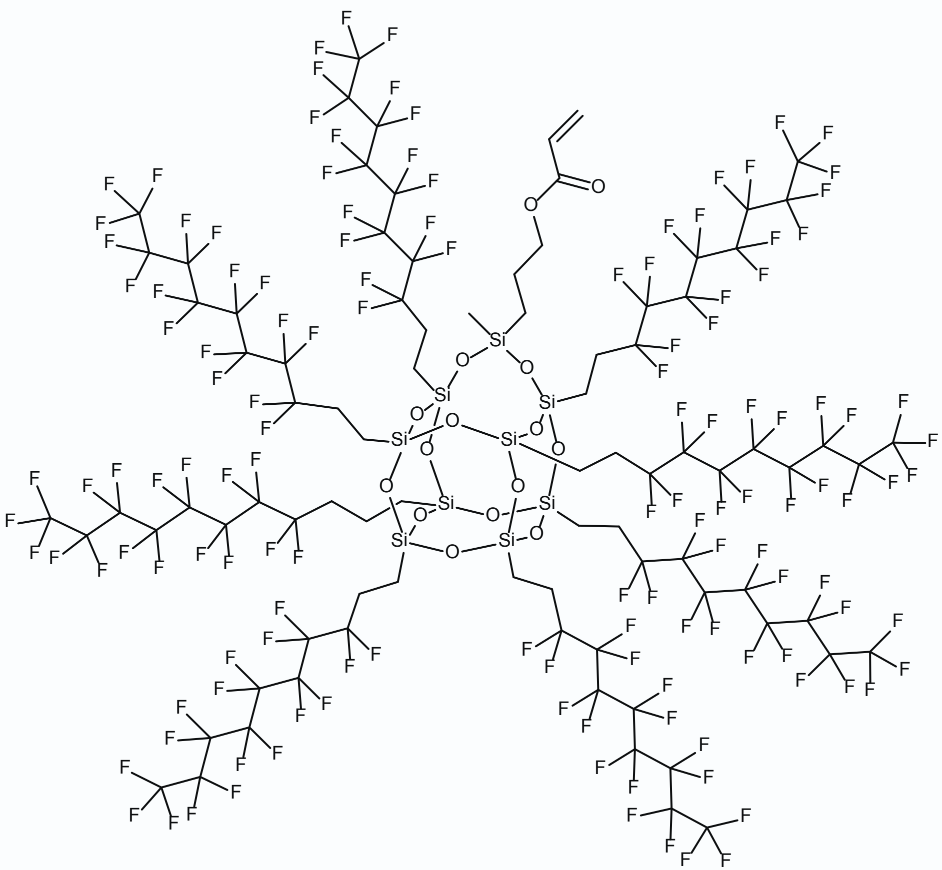
Ramirez, S. M.; Diaz, Y. J.; Campos, R. ; Stone, R.T.; Haddad, T.S.; Mabry, J.M., *J. Am. Chem. Soc.*, **2011**, *133*, 20084.

Edge Capping Reactions



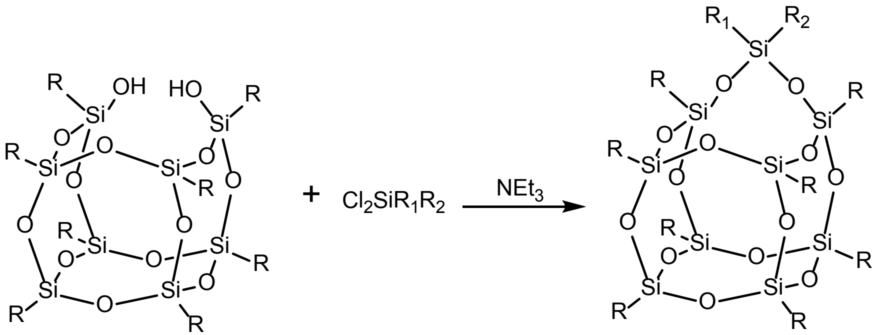
R = $CH_2CH_2(CF_2)_7CF_3$
R_1 = CH_3
R_2 = $CH_2CH_2CH_2$ OC(O)CHCH$_2$

- Edge capping reactions typically have 40-70% yield
- Main side product is starting material (recycled)
- Disilanol can revert back to closed cage during reaction
- Reactions take 5-10 minutes



Macromer/RBM = 4178 g/mol

Edge Capping Reactions



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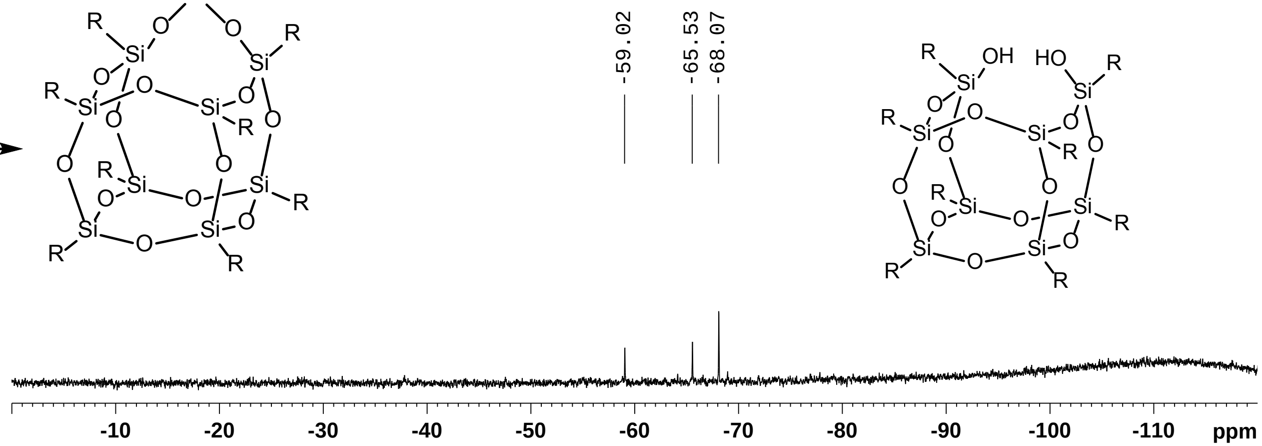
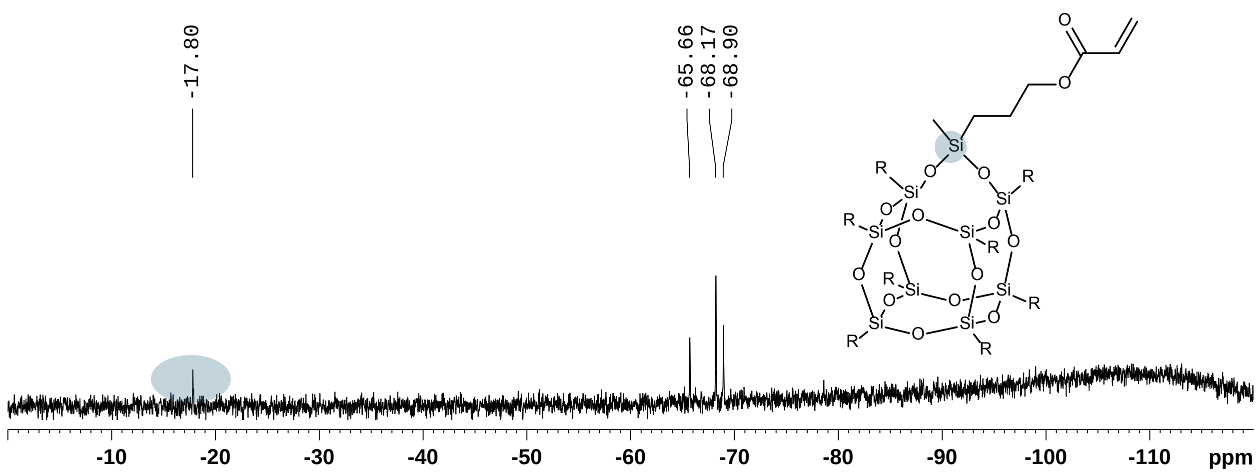
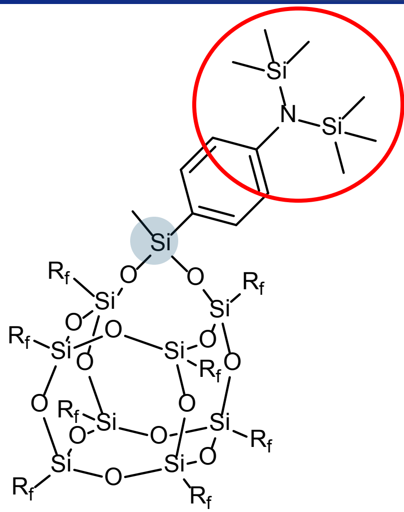


- Typically 40-70% yield
- Main side product is starting material (recycled), formed during base addition
- Disilanol can revert back to closed cage during reaction
- Reactions take 5-10 minutes
- Si ratio (1:2:2:4)
- **New Si peak!**

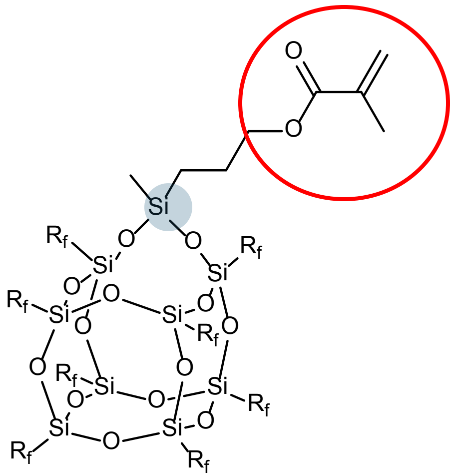


^{29}Si NMR taken in fluorinated solvent

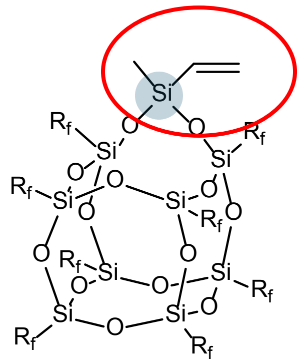
F-POSS Structures Synthesized



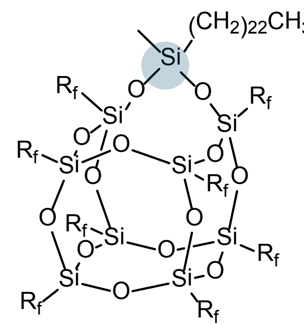
-29.5 ppm



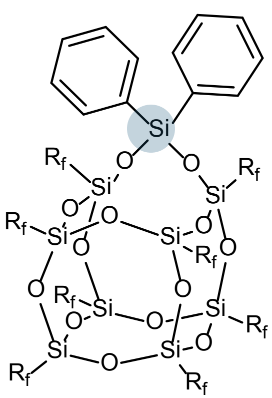
-17.8ppm



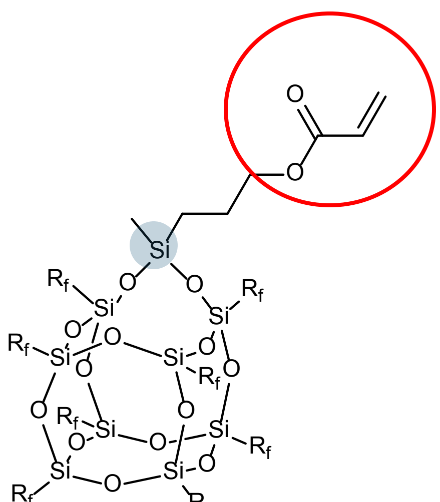
-32.1 ppm



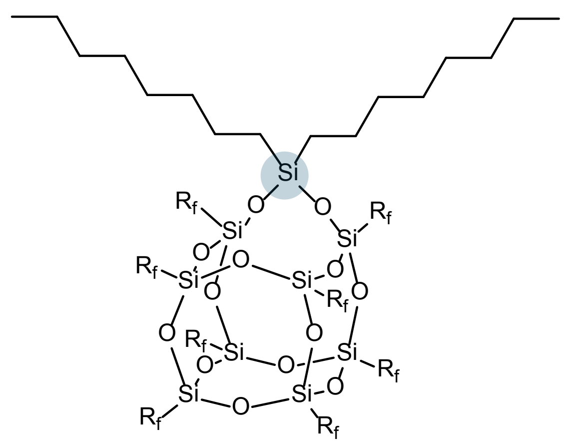
-17.8 ppm



-45.5 ppm



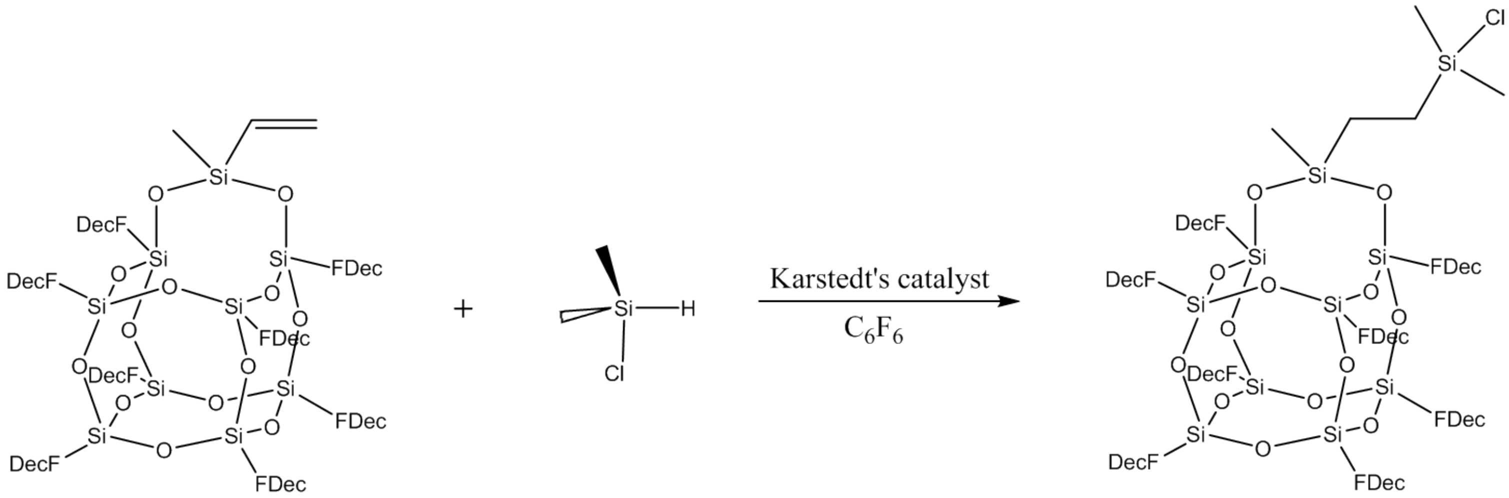
-17.1 ppm



-17.9 ppm

$R = CH_2CH_2(CF_2)_7CF_3$

F-POSS Silane Coupling Reaction

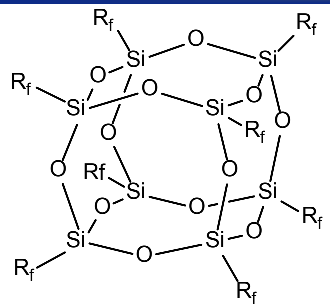


• Chlorosilyl-functional fluoroPOSS compound synthesized from the Pt(II) catalyzed hydrosilyation of vinyl-functional fluoroPOSS and dimethylchlorosilane

• Desired compound successfully synthesized in high yield

•Characterized by ^{1}H, ^{13}C, ^{19}F, and ^{29}Si NMR

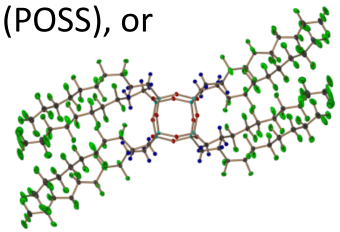
Introduction to F-POSS



R_f = -$(CH_2)_2(CF_2)_7CF_3$

(1,1,2,2-tetrahydroperfluorodecyl)$_8Si_8O_{12}$ Polyhedral Oligomeric Silsesquioxane (POSS), or fluorodecyl POSS

- hybrid organic-inorganic structure
- well-defined polyhedral architecture
- long-chain fluoroalkyl substituents on periphery of cage

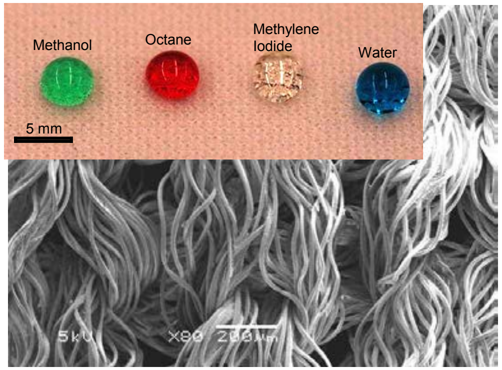


Due to its unique structure, fluorodecyl POSS has one of the lowest surface energies of any crystalline solid currently known

- fluorodecyl POSS	9.3 mN/m
- polytetrafluoroethylene	18-20 mN/m
- CF_3 monolayer	6.7 mN/m

Low surface energy and other unique properties of fluorodecyl POSS has enabled the development of various types of tunable non-wetting polymeric surfaces

Superhydrophobic/oleophilic dip-coated fabric
Tuteja *et al*, Science, **2007**, 318, 1618

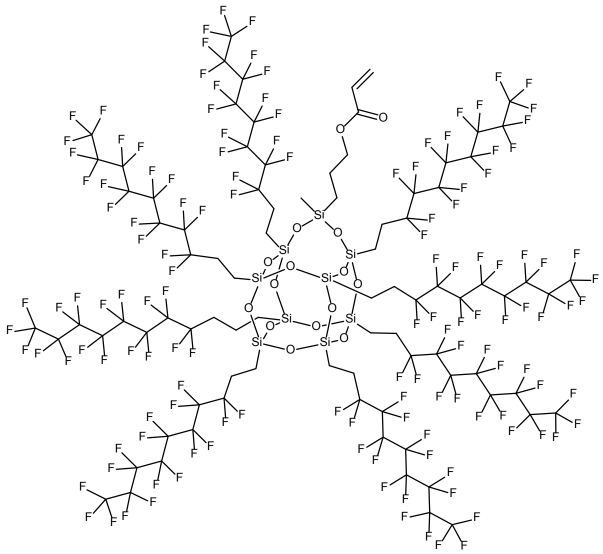


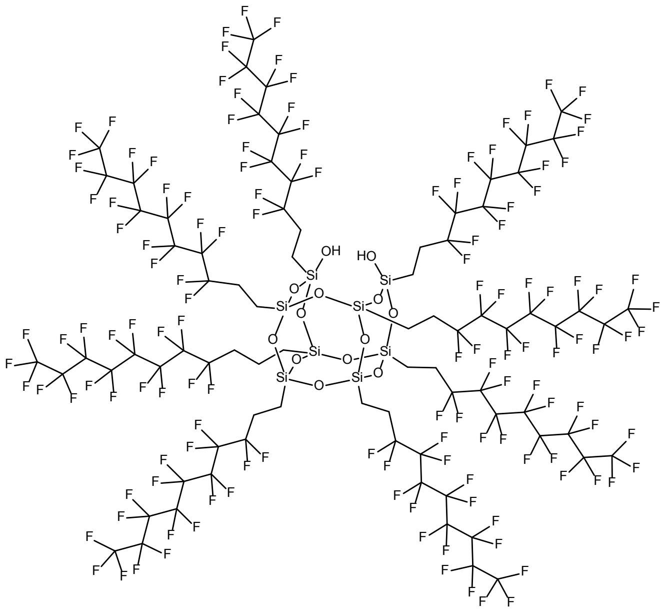
Superamphiphobic dip-coated fabric
Choi *et al*, Adv Mater, **2009**, 21, 2190

Superamphiphobic electrospun surfaces
Tuteja *et al*, PNAS, **2008**, 105, 18200

Contact Angle Measurements

- Non-wetting surfaces can be developed by a combination of three parameters
  - Chemical functionality (high fluorine content)
  - Roughness (micro- and nanoscale)
  - Surface Geometry (re-entrant curvature)
- *What type of influence will functional groups have on F-POSS surface properties?*
- *Solvent impact?*





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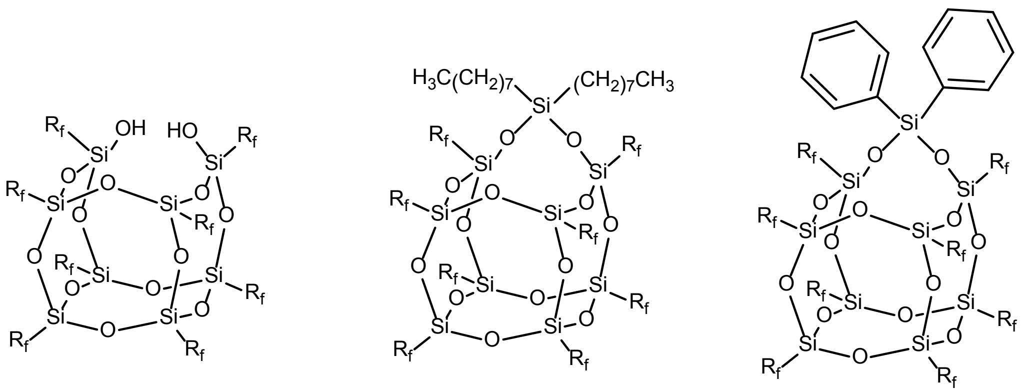
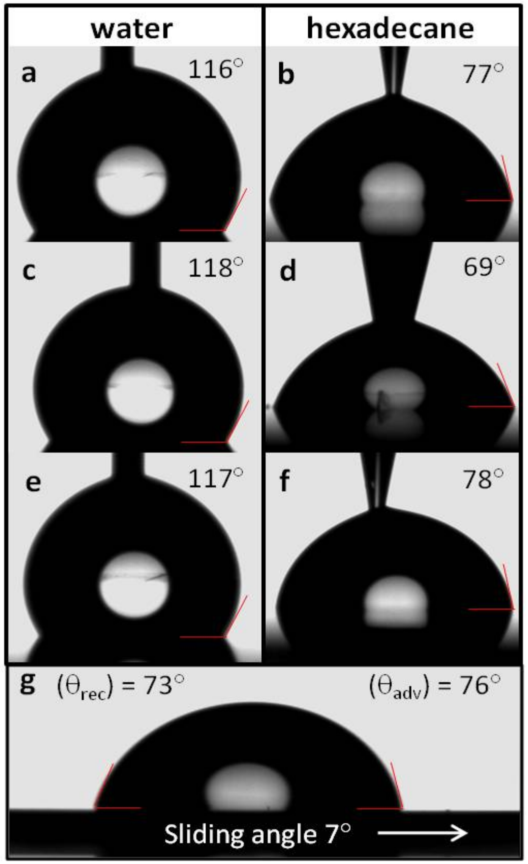
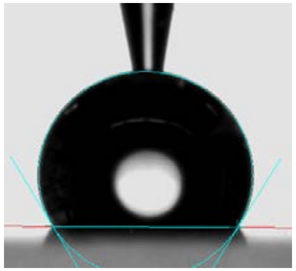




Static contact angles of Si wafer surfaces coated with compounds **disilanol** (a) and (b), **dioctyl** (c) and (d), and **diphenyl** (e) and (f). Image of hexadecane droplet (10 μL) rolling off surface coated with compound **diphenyl** (g).

Dynamic Contact Angle Measurements



Functional Group on F-POSS	*water*		*hexadecane*	
	(θ_{adv})	(θ_{rec})	(θ_{adv})	(θ_{rec})
F-POSS*	124 ± 0.5°	109.6 ± 0.7°	79.1 ± 0.4°	65.1 ± 0.5°
$Si\text{-}(OH)_2$	116.8 ± 0.4°	111 ± 0.6°	77.4 ± 0.4°	74.4 ± 0.8°
$Si\text{-}(CH_3)(CH{=}CH_2)$	116.2 ± 0.4°	100.6 ± 0.8°	78.4 ± 0.3°	70.6 ± 2.3°
$Si((CH_3)((CH_2)_3OC(O)CCH{=}CH_2)$	118.2 ± 1.0°	90.6 ± 1.0°	76.8 ± 0.3°	64.8 ± 1.0°
$Si\text{-}(CH_3)(\,(CH_2)_3OC(O)C(CH_3){=}CH_2)$	117.1 ± 0.6°	93.8 ± 1.5°	78.1 ± 0.4°	63.0 ± 1.2°
$Si\text{-}(CH_3)((CH_2)_{22}CH_3)$	117.9 ± 0.4°	96.9 ± 1.9°	78.0 ± 0.4°	16.2 ± 5.5°
$Si\text{-}(C_6H_5)_2$	116.2 ± 0.4°	110.5 ± 0.5°	76.0 ± 0.8°	73.2 ± 0.4°
$Si\text{-}((CH_2)_7CH_3)_2$	117.9 ± 0.5°	95.5 ± 0.4°	69.1 ± 1.2°	23.1 ± 1.2°

Samples (10 mg/mL) were spin casted on oxygen-plasma cleaned Si wafers at 900 rpm for 30 seconds. Contact angle measurements were run in triplicate. Surface roughness < 5nm (AFM and Optical Profilometry).

*Chhatre, S. S.; Guardado, J. O.; Moore, B. M.; Haddad, T. S.; Mabry, J. M.; McKinley, G. H.; Cohen, R. E. *ACS Appl. Mater. Interfaces* **2010**, *2*, 3544.

Free Radical Polymerization

A (Monomer)

Polymer AAAAAAAAAAAA

$-\!\!\left(A\right)\!\!-_n$

A + B (Monomers)

Copolymer ABAABABAB

AAAAAAABBBBBBBB

Initiation

$$\text{Initiator} \xrightarrow{k_d} 2\,\text{I}\bullet$$

$$\text{I}\bullet + \text{Monomer} \xrightarrow{k_i} P_n\bullet$$

Propogation

$$P_n\bullet + \text{Monomer} \xrightarrow{k_p} P_n\bullet$$

Termination

$$2\ \text{Radicals} \xrightarrow{k_t} \text{Polymer}$$

-Monomers make polymers

-Multiple different types of monomers in a polymer make copolymers

-Choosing a type of monomer will decide what type of polymer you have

-trash bags, cotton, paint, DNA, protein, plastic bottles, etc…..most things you use in your life

Free Radical Polymerization

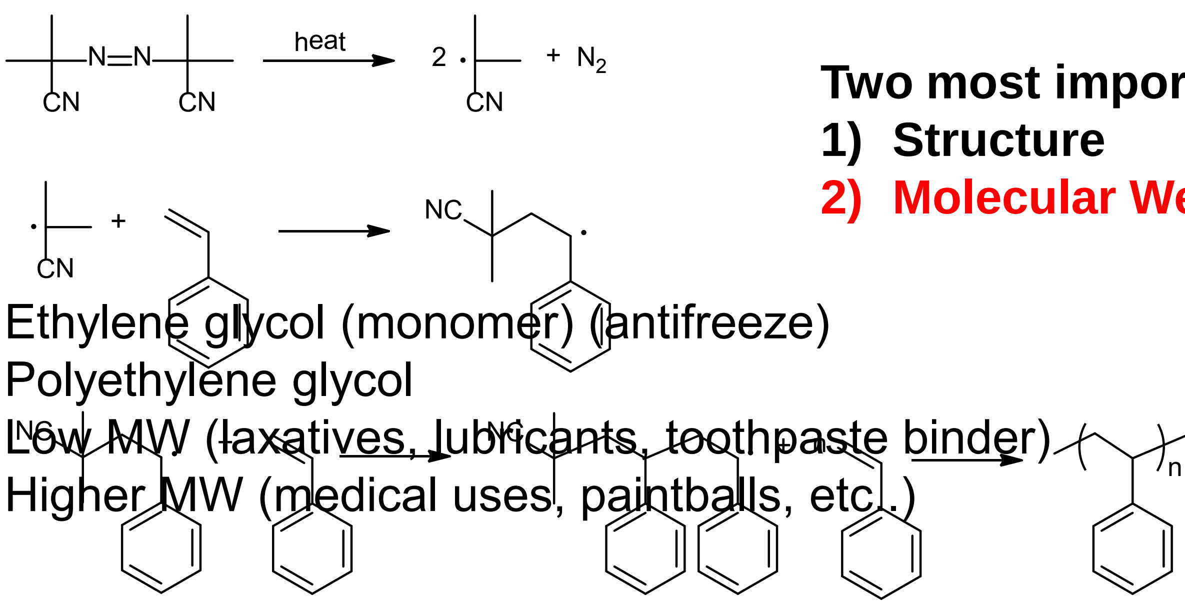
Initiation: Initiator $\xrightarrow{k_d}$ 2 I•

I• + Monomer $\xrightarrow{k_i}$ P_n•

Propogation: P_n• + Monomer $\xrightarrow{k_p}$ P_n•

Termination: 2 Radicals $\xrightarrow{k_t}$ Polymer

Example: Polystyrene



Ethylene glycol (monomer) (antifreeze)
Polyethylene glycol
Low MW (laxatives, lubricants, toothpaste binder)
Higher MW (medical uses, paintballs, etc.)

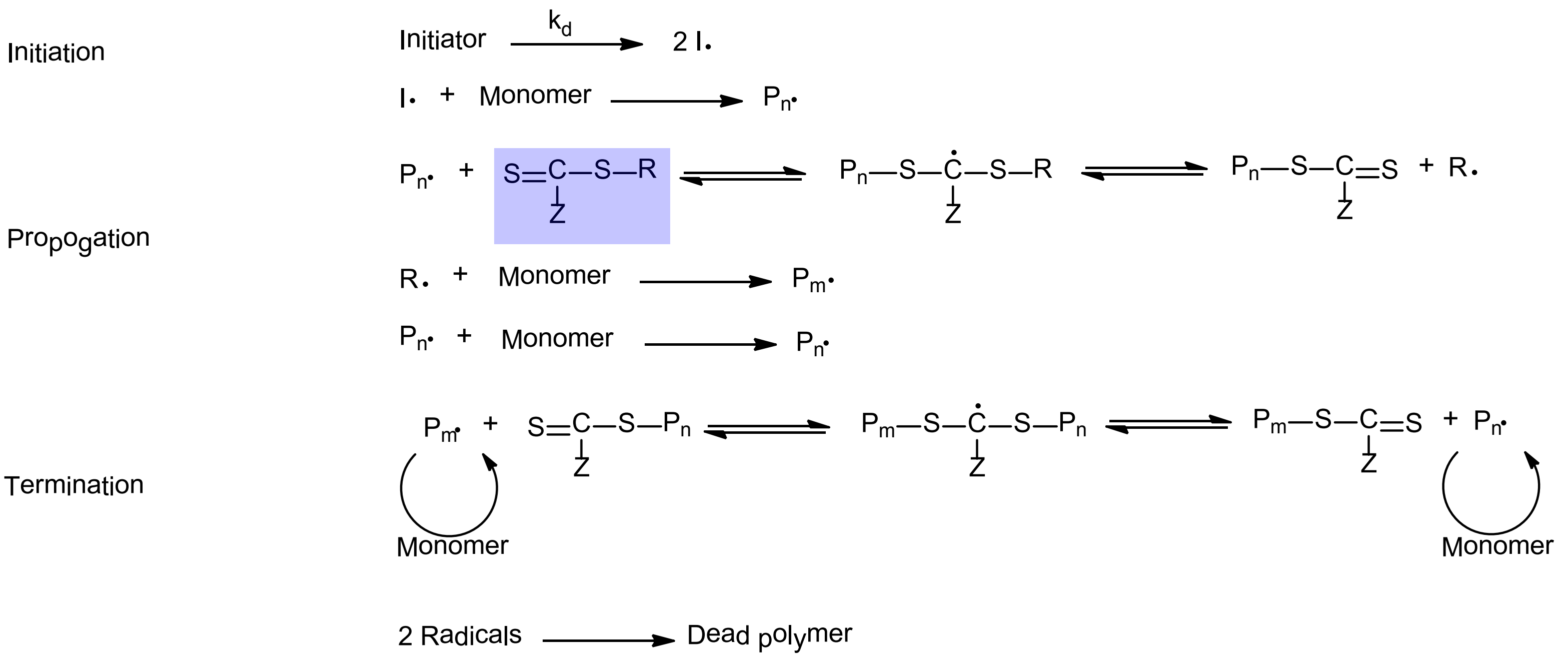
- Standard polymerization method
- Simple, cheap, easy
- Does not allow for much control
- No block copolymers
- molecular weight difficult to control

Two most important factors for polymers:

1) Structure

2) Molecular Weight

Reversible Addition-Fragmentation chain Transfer (RAFT) polymerization



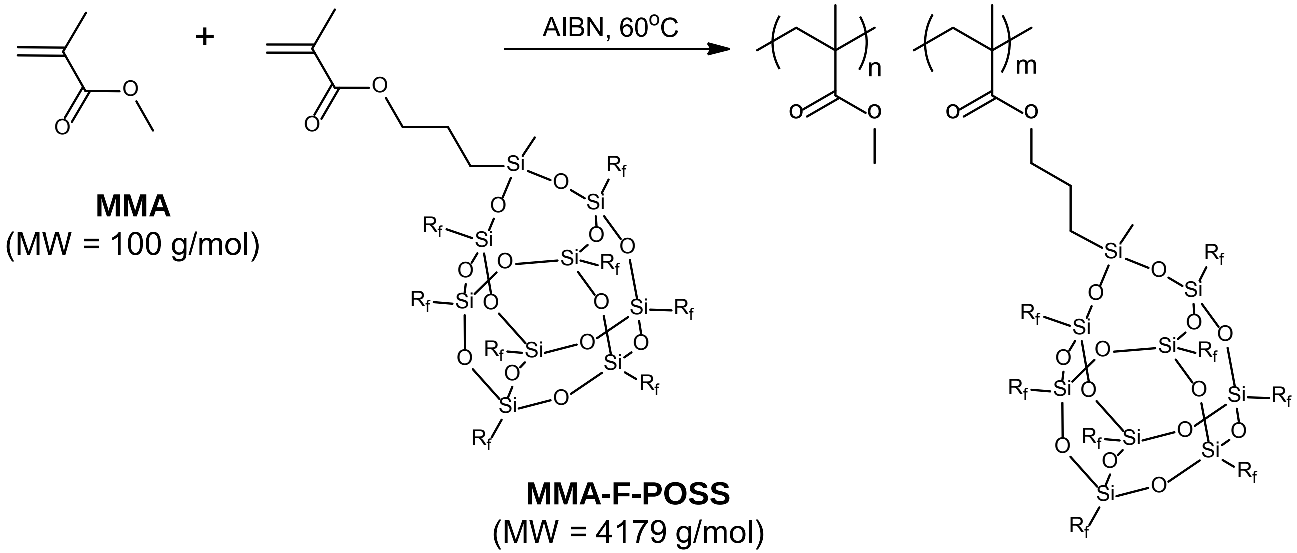
Chain Transfer Agent

RAFT Polymerization

- Controlled polymerization
- Allows for block copolymers
- Tune molecular weight

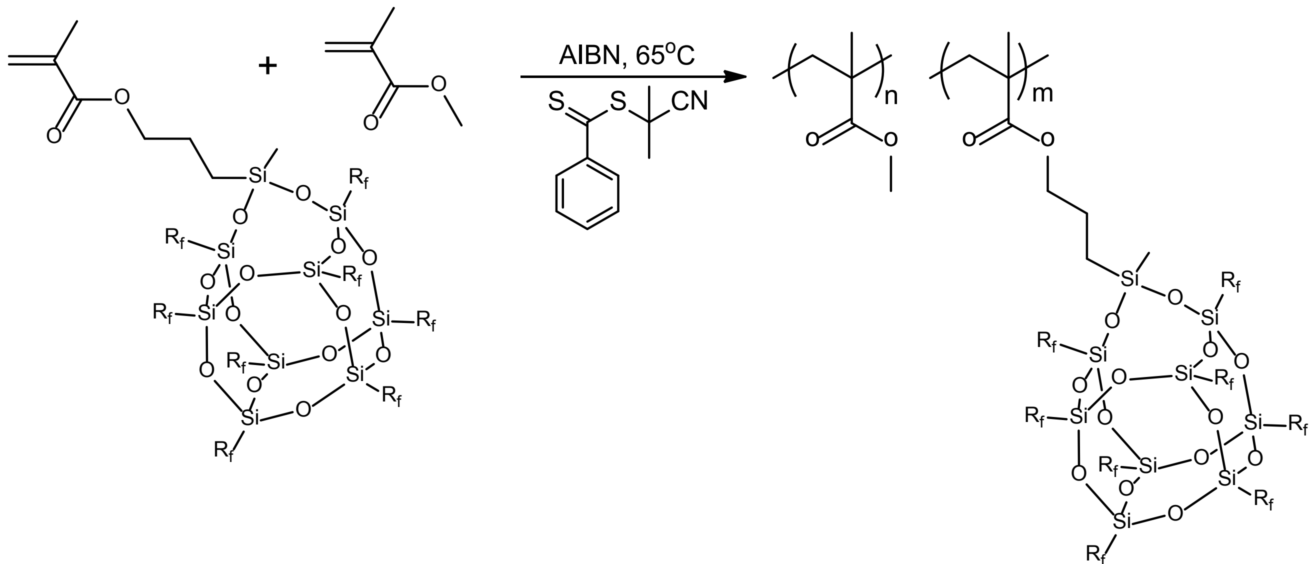
Chiefari, J.; Chong, Y. K.; Ercole, F.; Krstina, J.; Jeffery, J.; Le, T. P. T.; Mayadunne, R. T. A.; Meijs, G. F.; Moad, C. L.; Moad, G.; Rizzardo, E.; Thang, S. H. Living Free Radical Polymerization by Reversible Addition-Fragmentation Chain Transfer: The RAFT Process. *Macromolecules* 1998, *31*, 5559−5562

Free Radical Copolymerizations



- Standard free radical polymerization of methyl methacrylate and MMA-F-POSS monomers:
  - F-POSS monomer is active towards polymerization
  - F-POSS incorporation (1-20% by weight)
  - Molecular weights range from 10-50,000 g/mol
  - Higher F-POSS and polymerization causes problems

RAFT Copolymerizations



- Controlled/"living" free radical polymerization of methyl methacrylate, CPD, and MMA-F-POSS monomers:
  - Promising results with molecular weights ranging from 20-40,000 gm/mol
  - Narrow polydispersity indices (1.04-1.1)

Summary

- Structures were demonstrated to be reactive towards a variety of dichlorosilanes
- Solubility of F-POSS compounds were shown to be influenced by chemical functionality
- Functionality was shown to be influential on contact angle measurements
- Currently working on other monomers and polymers for F-POSS
- F-POSS compounds have a near limitless potential in producing a variety of new hydrophobic, oleophobic, or ominiphobic polymer composites.
  - Reaction mechanisms, polymer composites, block copolymers, etc….

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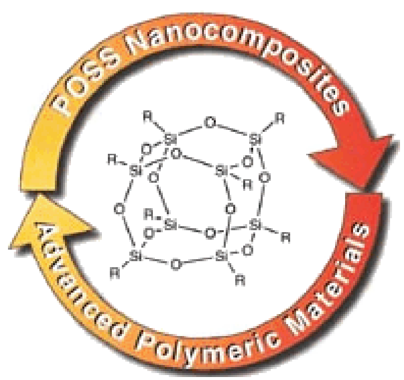
Ms. Yvonne Diaz
Mr. Raymond Compos



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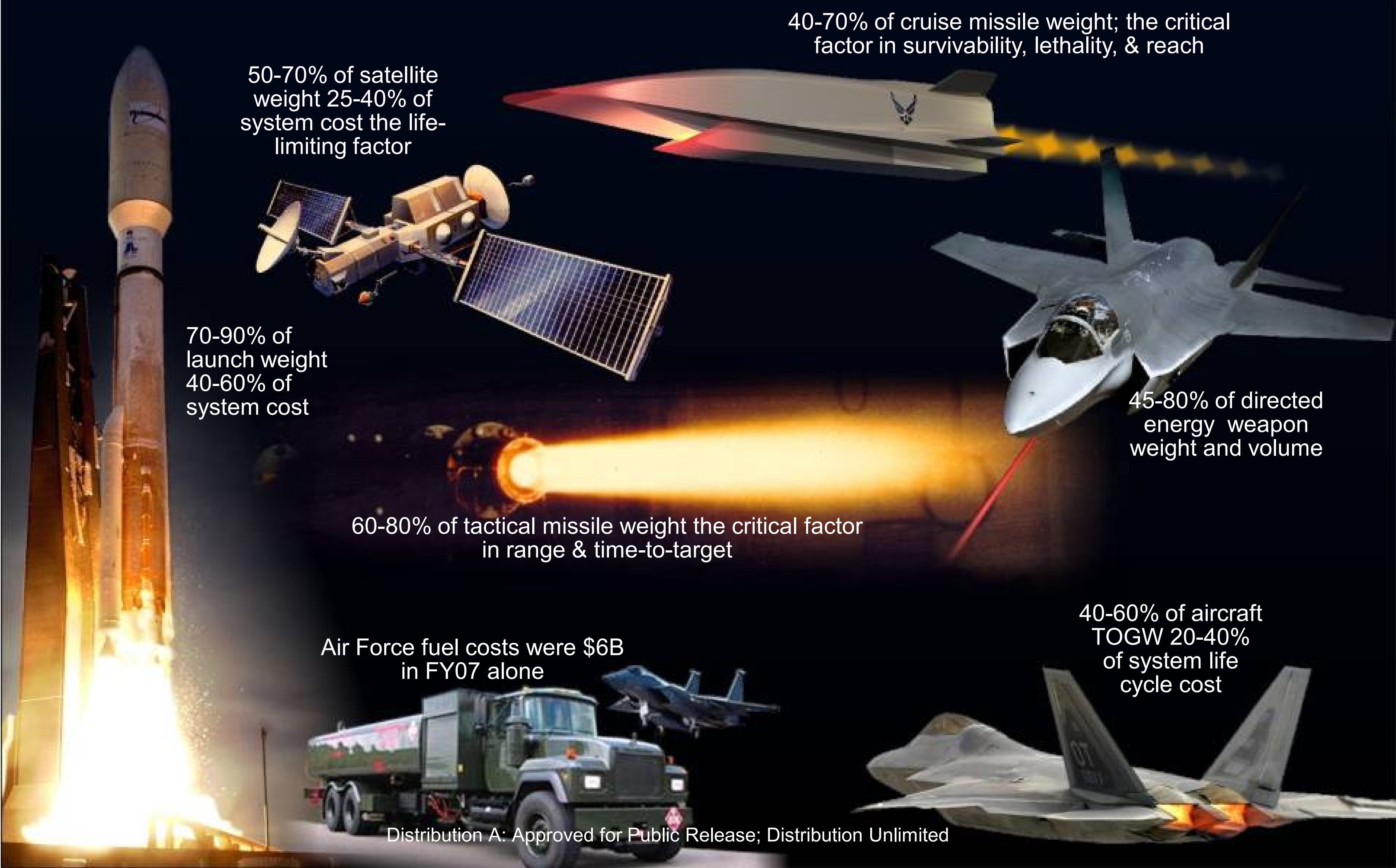
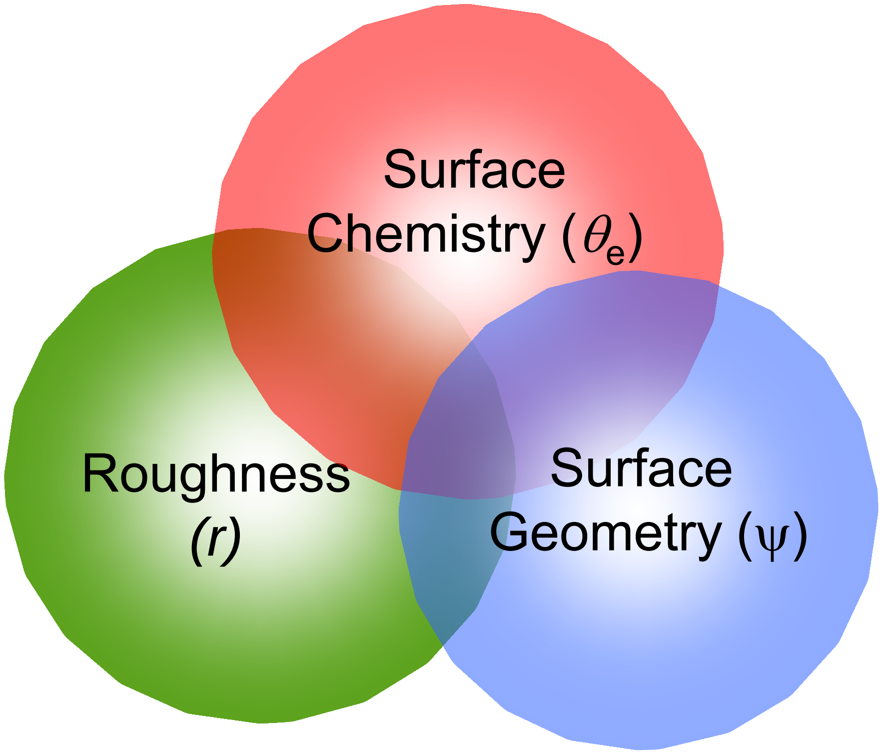
Air Force Research Laboratory, Propulsion Directorate

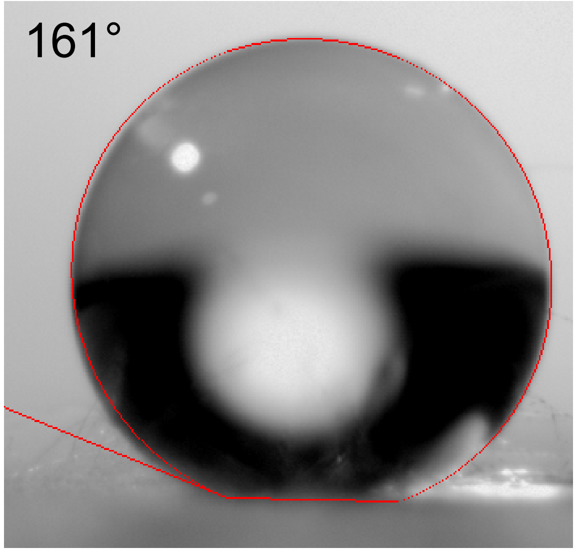
Propulsion & Power are Important!



Designing Superoleophobic Surfaces

- **Goal: a design framework for constructing super-repellent surfaces**
- **Demonstrated electrospun mats (single step process)**
- **Three key ingredients**





Superhydrophobic

$\theta > 150°$

PMMA + 44 wt% POSS

electrospun coating (beads on a string) morphology